



NBS TECHNICAL NOTE 744

Disclosures On:

New Syntheses of Perfluorostyrene
and Other Highly Fluorinated Derivatives

Temperature-, Radiation-, and Vacuum-Resistant Magnetic Tape

Conductometric Titration Cell

Mill Work Positioner

Process for Fabricating Superconducting Microbridges

Apparatus for Displaying Average Wind Vane or Other Shaft Position

Document Numbering Machine Responsive to a Staple
in a Print Area for Printing in an Alternate Area

U.S.
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David Robbins and Alvin J. Englert, Editors

Patent Adviser's Office
National Bureau of Standards
Washington, D.C. 20234

NBS Technical Notes are designed to supplement the Bureau's regular publications program. They provide a means for making available scientific data that are of transient or limited interest. Technical Notes may be listed or referred to in the open literature.



U.S. DEPARTMENT OF COMMERCE, Peter G. Peterson, Secretary

NATIONAL BUREAU OF STANDARDS, Lawrence M. Kushner, Acting Director,

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PREFACE

This NBS Technical Note is one of a continuing series entitled "Disclosures on" This series consists of short descriptions of recent developments from the Department of Commerce primary operating units, departmental staff offices, and their contractors. It is believed that these developments have important commercial applications. Unless otherwise stated in the description of a development, the property rights therein have been retained by the United States as represented by the Secretary of Commerce.

Previous NBS Technical Notes in this series are NBS Technical Notes 237, 253, 263, 282, 287, 295, 437, 440 and 536.

David Robbins
Patent Adviser

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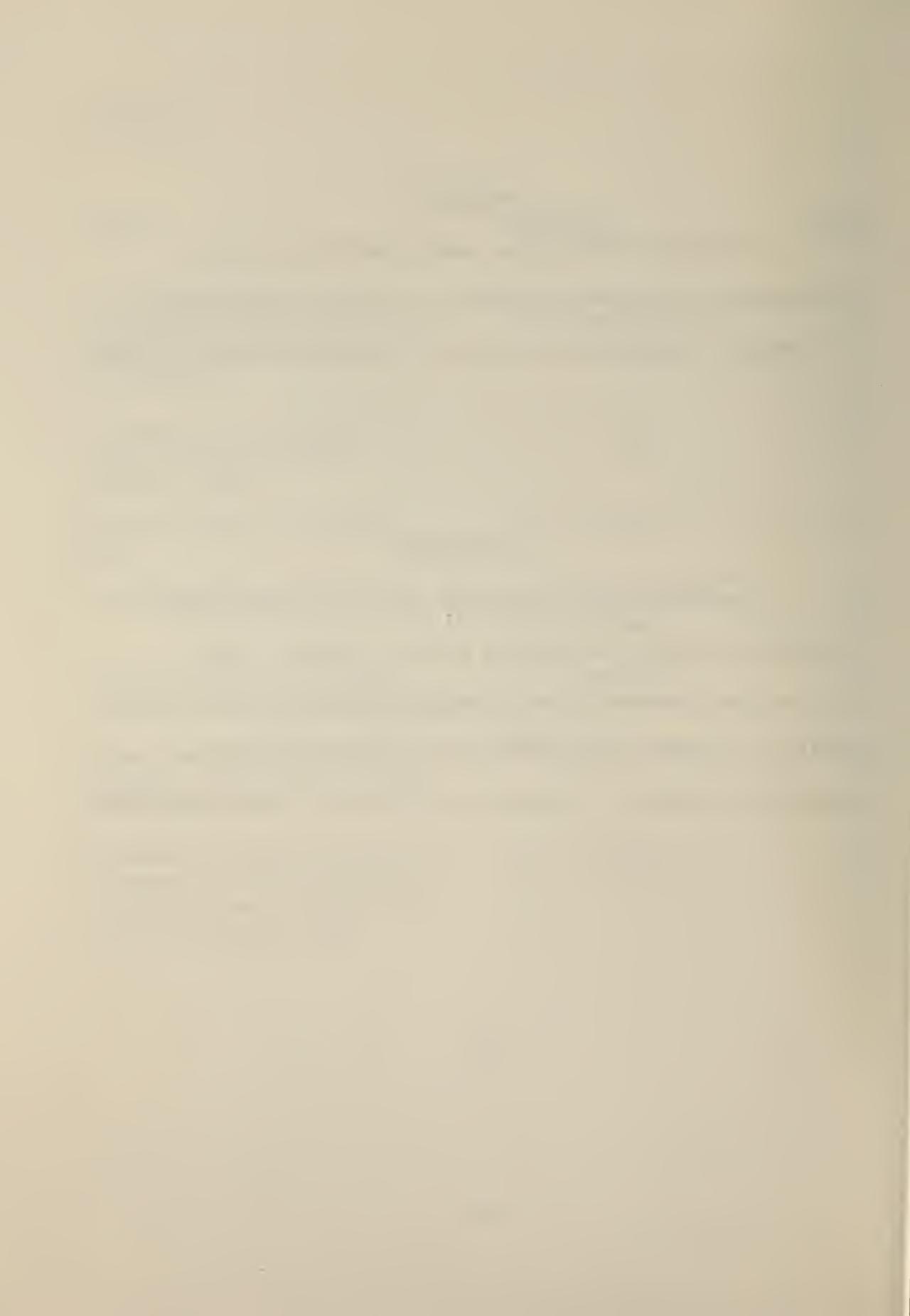
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ABSTRACT

This Note describes and illustrates seven developments that are believed to embody interesting and unusual solutions to current problems in their fields.

KEY WORDS

Conductometry; decoding matrix; fluoroolefins; integrator; magnetic sensing probe; magnetic tape; microbridge; numbering machine; perfluorostyrene; shaft position encoder; staple detector; superconducting; synthesis, chemical; titration; wind vane; work positioner.



NEW SYNTHESES OF PERFLUOROSTYRENE AND OTHER HIGHLY FLUORINATED DERIVATIVES

L. A. Wall, J. M. Antonucci and D. W. Brown

U. S. Pat. No. 2,874,197 to Dixon discloses that certain fluoroolefins are capable of reacting with organolithium compounds by an addition-elimination process that results in the formation of new fluoroolefins and lithium fluoride. The general reaction scheme is



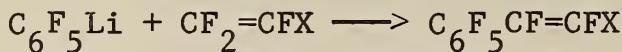
where X_1 and X_2 are both electronegative atoms or groups and Y is F or a fluoroalkyl group.

The application of this method to the synthesis of perfluorostyrene by the reaction of perfluorophenylolithium and a moderate excess of tetrafluoroethylene at low temperatures (-80 to 20° C) failed to yield any of the desired monomer. Instead a very complex mixture of solid products was isolated. It is anticipated that the desired monomer will be useful for the preparation of new thermally stable polymeric materials.

Careful analysis of all the chief products and reactants in a series of investigations enabled us, however, to develop a process capable of producing the desired monomer. By employing a very large excess of tetrafluoroethylene (threefold or greater) and controlled reaction times at low temperatures, it was possible to form perfluorostyrene in fair yields (10-20%). The formation of perfluorostyrene and the other monomers by this reaction was followed most conveniently by gas-liquid chromatography. From these studies, it was found that the yield of monomer is quite time dependent, reaching a maximum at a certain reaction time and decreasing thereafter. As expected, the optimum reaction time varies with the temperature of the reaction and with the specific reaction mixture employed.

General Method of Synthesis

The general scheme is



where X = F, Cl; H, CF₃

Synthesis of Perfluorophenyllithium

Direct Metallation. Pentafluoriodobenzene in ether was lithiated at -20 to 0° C by use of a lithium dispersion in paraffin. The technique employed was essentially that used to lithiate conventional halobenzenes.

Exchange Reaction. Pentafluorobenzene in ether was treated with an organolithium reagent, such as methyl-lithium, at -80° C to give the desired perfluorophenyl-lithium. The technique employed was essentially that reported by Tamborski *et al.*, J. Org. Chem. 29, 2385 (1964).

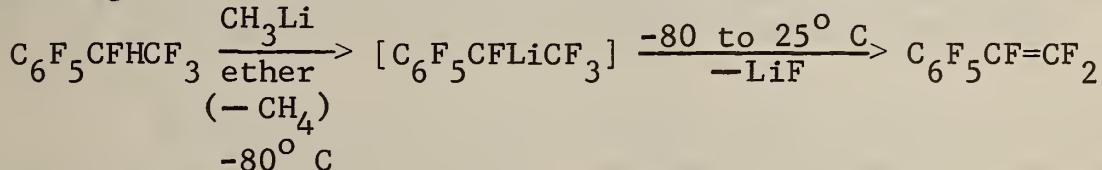
Synthesis of Perfluorostyrene

One mole of perfluorophenyllithium in 15 liters of ether was prepared in a 30-liter steel bomb maintained at -80° C. To this was added carefully 15 moles of tetrafluoroethylene. The bomb was sealed and then agitated for 3 to 5 days at -20° C. The reaction mixture was treated as follows at the conclusion of the reaction: any unreacted perfluorophenyllithium was hydrolyzed, the unreacted tetrafluoroethylene was removed and collected. The ether solution was then worked up in the usual way to yield 20% perfluorostyrene, b.p. 122° C.

The same technique also proved successful in the reaction of trifluoroethylene with perfluorophenyllithium to give β-hydroheptafluorostyrene. Other successful syntheses included the reaction of perfluorophenyllithium with chlorotrifluoroethylene and perfluoropropylene to give β-chloroheptafluorostyrene and perfluoro-β-methylstyrene, respectively.

Special Method of Synthesis of Perfluorostyrene

A related but somewhat different synthesis of perfluorostyrene was discovered in the course of these investigations and involves the dehydrofluorination of α -hydrononafluoroethylbenzene (Wall *et al.* U. S. Pat. No. 3,265,746) with methylolithium at -80°C , followed by warming to 25°C . The reaction sequence is believed to be

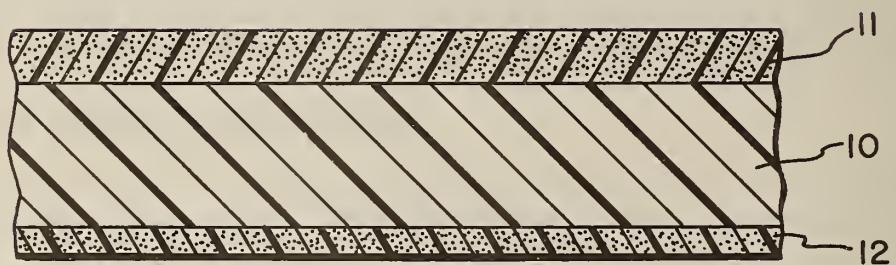


-80°C

Yields of perfluorostyrene of the order of 85 to 90% can be realized by this method.

TEMPERATURE-, RADIATION-, AND VACUUM-RESISTANT MAGNETIC TAPE

K. F. Plitt



TEMPERATURE-, RADIATION-, AND VACUUM-RESISTANT MAGNETIC TAPE

K. F. Plitt

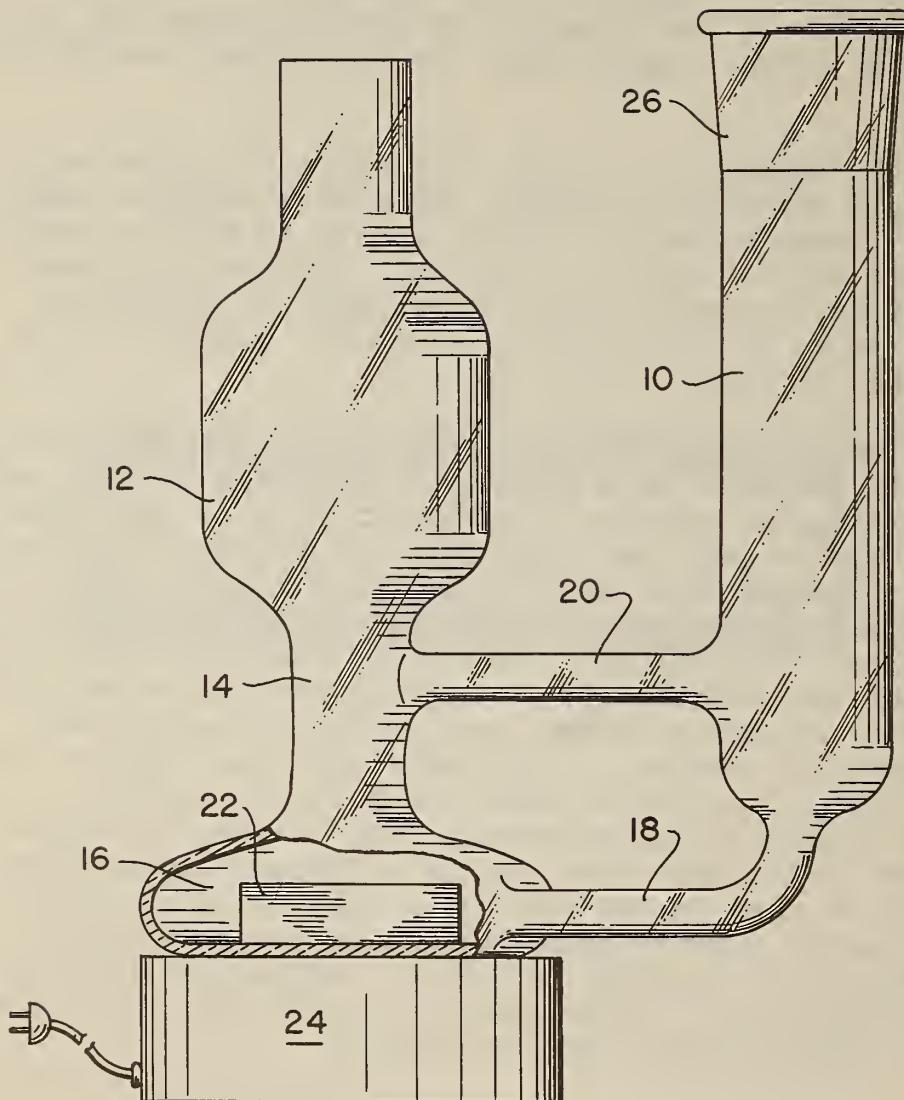
Satellite tape recorders frequently fail because the magnetic tape sticks to a head surface. This is apparently due to the transfer of material from the tape to the head surface until that surface adheres easily to the tape.

In addition to a tape that will not contaminate head surfaces, a satellite recorder requires a magnetic tape that does not change properties when exposed to hard vacuum and has a high resistance to radiation. Further, for some applications, the tape should be capable of operating properly after sterilization by gaseous chemicals or by heating for 24 hours at 140° C.

The tape described in this note meets these requirements. It consists of a base film 10 coated on each side with a different coating. The base film is a polyimide material chosen for its stability, temperature resistance, mechanical properties, and freedom from liquid plasticizers and volatile constituents. The coating 11 on the front side of the base film consists of a binder, magnetic iron oxide, and conductive carbon. It is applied from solution, dried, and cured. Suitable binders are polyimide resins and polyamide-imide resins. Other materials that may be used include high temperature resistant polymers such as polybenzimidazoles and pyrrones. The coating 12 on the back side of the base consists of a binder, preferably the same polymer used in the coating on the front side, conductive carbon, and in some cases powdered polytetra-fluoroethylene. Coating 12 reduces cupping of tape during curing and provides lubrication for tapes that are to be used in "endless-loop" tape recorders.

CONDUCTOMETRIC TITRATION CELL

T. B. Hoover



CONDUCTOMETRIC TITRATION CELL

T. B. Hoover

Ten microequivalents or less of a specimen dissolved in 25-50 ml of solvent can be titrated using this cell. The titration, which may be performed in an inert atmosphere, is monitored by measuring the solution conductivity as the reagent is added. The solution is stirred magnetically and the electrodes are positioned away from the stirrer to avoid current induction by the rotating magnetic field.

As shown in the figure, the cell consists of an electrode chamber 10 and a flask 12 with a neck 14 and a stirring chamber 16. A feed arm 18 extending tangentially from the stirring chamber 16 is connected to the bottom of the electrode chamber 10 and a return arm 20 is connected to the flask neck 14. A magnetic stirring bar 22 introduced into the stirring chamber 16 through the neck 14 is driven by a rotating magnet drive 24. The solution is thus centrifugally stirred and circulated through the cell. The electrode circuit or "loop" (not shown), being outside the fringing stirrer field, avoids the induction of extraneous currents.

The electrode chamber top has a standard taper joint 26 for receiving interchangeable dipping electrodes of different cell constants appropriate to the solvent-reagent system used. The flask 12 may be provided with a serum cap (not shown) through which the titrant and a purge or filling gas for controlling the cell atmosphere may be introduced by hypodermic needles.

MILL WORK POSITIONER

J. M. Holloway and K. H. Gebert

This device may be used to position work on a milling machine.

In the figures bolt 10 passes through post 11. Machine nut 12 is threaded on one end of the bolt and T-nut 13 is threaded on the other end. Stop rod 14 is made off set and has one end located in one of the holes 15 in the post. Instead of 14, two straight arms and a swivel clamp may be used. When this arrangement is used, one of the arms is located in a fixed position between the clamp and post 11, while the other arm is located in the clamp. By proper adjustment of the clamp, an end of the second arm may be moved horizontally, vertically, or along a desired angle.

In a typical operation, T-nut 13 is positioned in a slot in the table of the milling machine. The T-nut is tightened. A piece of work is placed in a vise on the table. Stop rod 14 is then positioned in the hole in post 11, corresponding to the height of the work, and the rod is rotated to make fine adjustments in height, if required, before it is clamped by the related screw 16. The piece of work is then moved horizontally in the vise to engage the end of stop rod 14 and is thereby positioned precisely on the milling machine.

MILL WORK POSITIONER

J. M. Holloway and K. H. Gebert

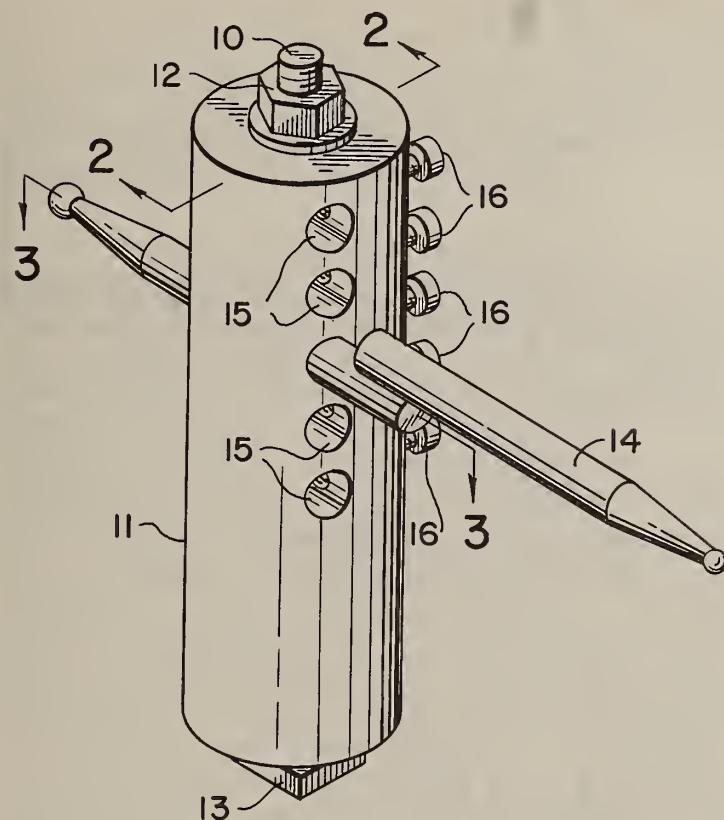


FIG. 1

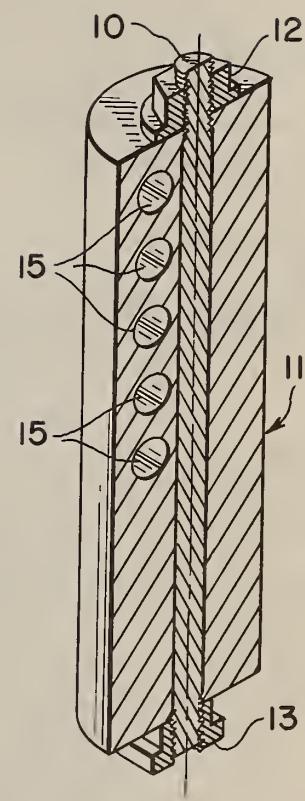


FIG. 2

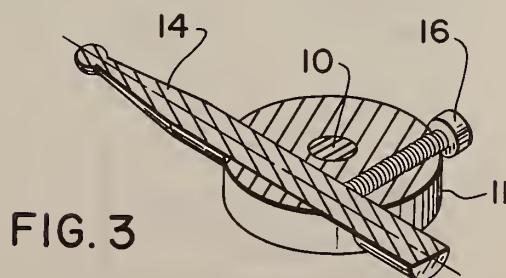
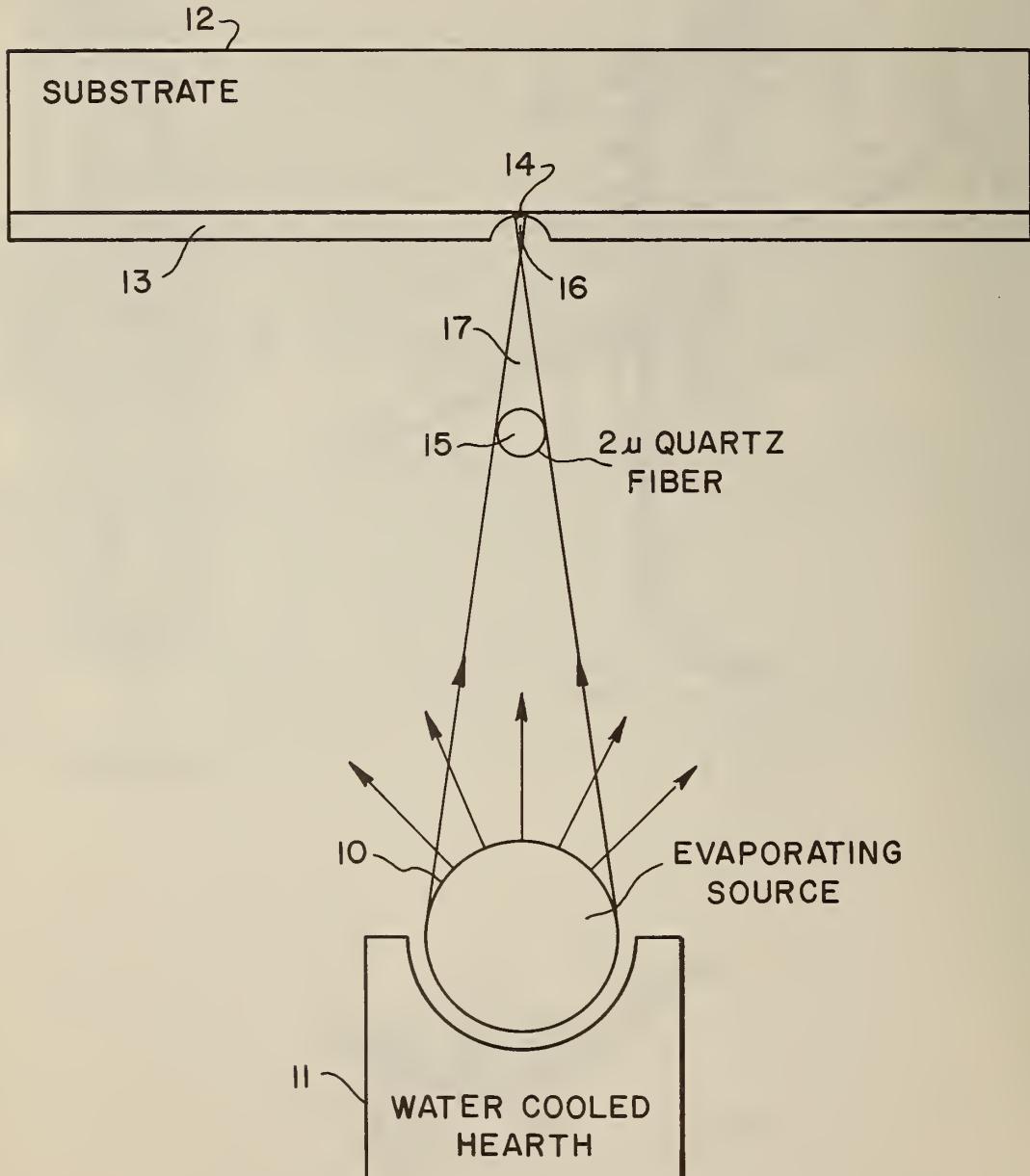


FIG. 3

PROCESS FOR FABRICATING SUPERCONDUCTING MICROBRIDGES

L. O. Mullen



PROCESS FOR FABRICATING SUPERCONDUCTING MICROBRIDGES

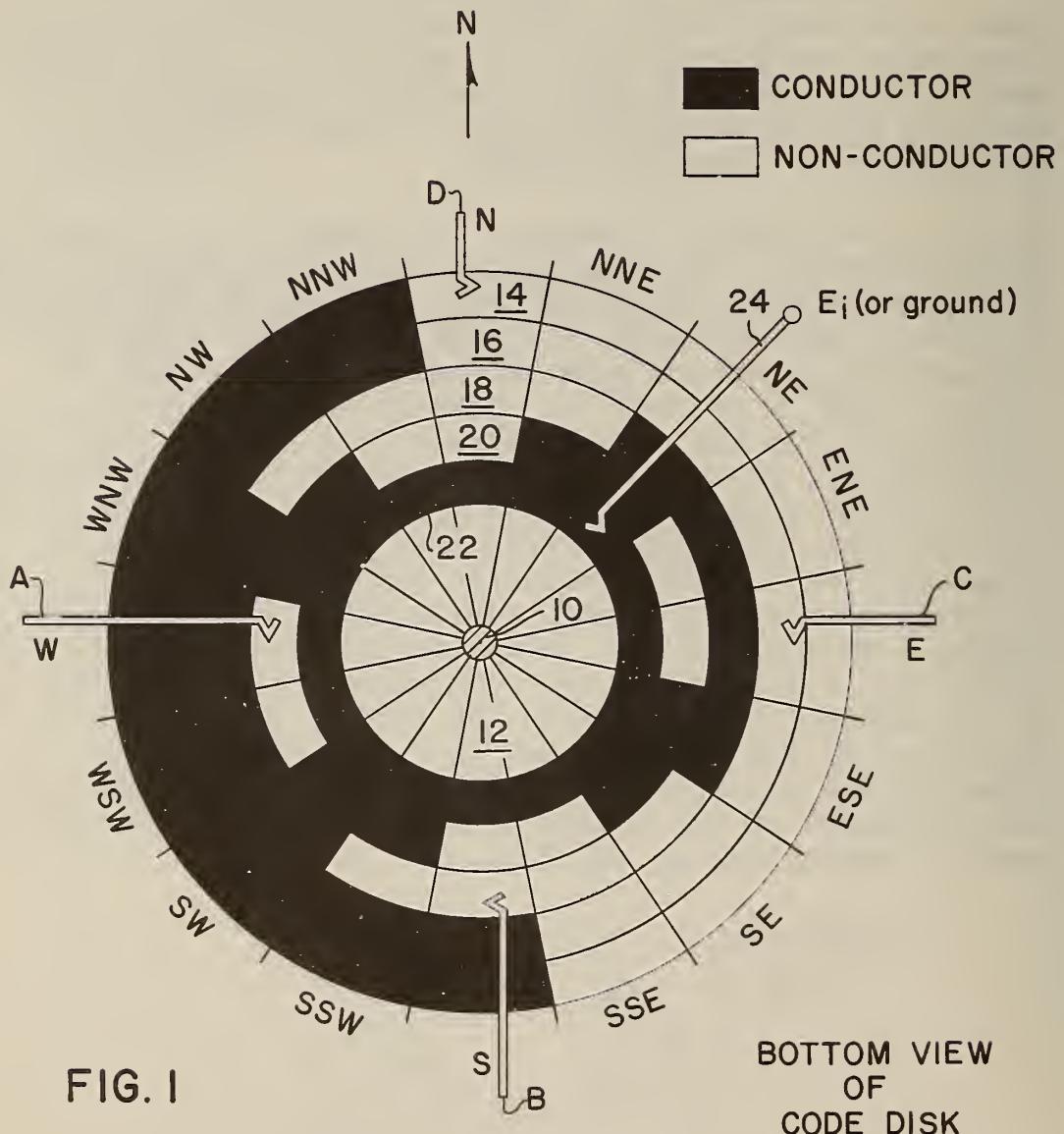
L. O.Mullen

The essence of a superconducting microbridge is a controlled constriction of a strip of superconducting thin film where the conductivity is weakened in a well-defined area. This note describes a process for precisely controlling the width and thickness of the film in the constriction or metallic bridge.

The structure shown in the figure is positioned in a suitable vacuum. An evaporating source 10 comprises a ball of superconducting material, such as niobium, and is placed in a water cooled hearth 11. The source is heated by a conventional electron beam arrangement, not shown, to between 3100° K to 3500° K. Some of the molecules, evaporated from the source, travel in a straight line toward substrate 12, which is preferably sapphire for niobium, and a thin film 13 is deposited on the surface of the substrate. The thickness and width of the film deposited along a selected line to form a metallic bridge 14 is controlled by properly positioning a thin quartz fiber 15. (The fiber may be 2 microns thick.) More specifically, the width and thickness of the bridge are controlled by the penumbra 16 of the shadow 17 produced by the fiber. The width and thickness of the penumbra are in turn controlled by the diameter and position of the fiber 15 from substrate 12. The thickness of the bridge also depends upon the time and rate of deposition of the film.

APPARATUS FOR DISPLAYING AVERAGE WIND VANE OR OTHER SHAFT POSITION

H. H. Crouser



APPARATUS FOR DISPLAYING AVERAGE WIND VANE OR OTHER SHAFT POSITION

H. H. Crouser

This device encodes instantaneous shaft position in a cyclic bit code having equal on-and-off distributions and time integrates each bit. If the integral exceeds a threshold, the bit is fed to a code converter which drives a display unit. The device is particularly useful in reporting average wind directions from a remote battery-powered weather station.

As shown in Fig. 1 the fluctuating wind vane or other shaft 10 is equipped with a cyclic code disk 12. The first and second outer rings 14 and 16 of the disk each have a 180° arcuate metal segment. The third ring 18 has two evenly spaced 90° segments and the fourth ring 20 has four 45° segments. For greater shaft position resolution there could be provided a fifth ring with eight 22.5° segments, and so on. The continuous inner band 22 is the common circuit terminal energized with voltage E_i from brush 24. The voltage is picked up from the rings 14, 16, 18 and 20 by brushes D, C, B and A respectively. For ease of construction these brushes are positioned at 90° intervals around the disk 12. The resulting cyclic code of the sixteen wind directions is as follows.

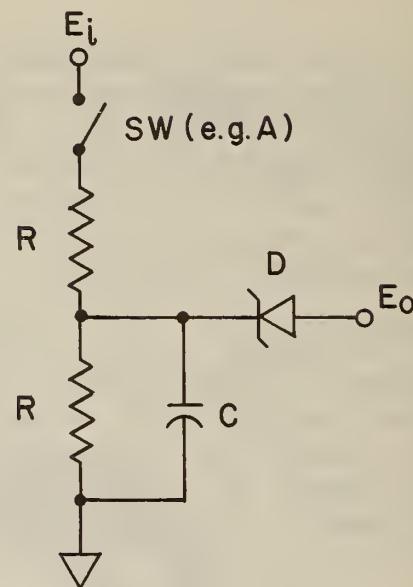
N	0000	S	0011
N NE	1000	S SW	1011
NE	1100	SW	1111
E NE	0100	W SW	0111
E	0110	W	0101
E SE	1110	W NW	1101
SE	1010	NW	1001
S SE	0010	N NW	0001

APPARATUS FOR DISPLAYING AVERAGE WIND VANE OR OTHER SHAFT POSITION

H. H. Crouser

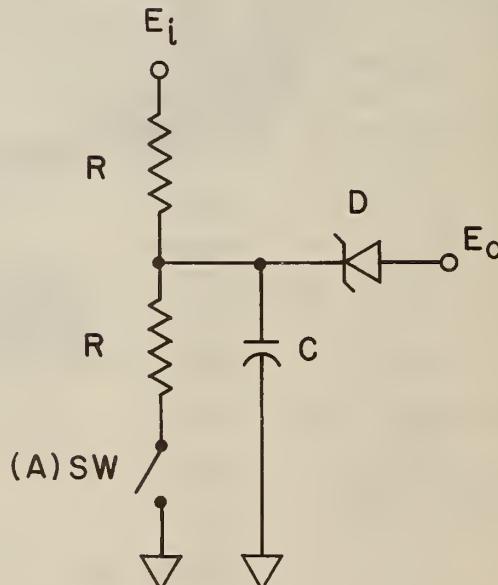
<u>SW</u>	E_o
OPEN	0
CLOSED	$E_i/2$
50% CLOSED	$E_i/4$

FIG. 2



<u>SW</u>	E_o
OPEN	E_i
CLOSED	$E_i/2$
50% CLOSED	$0.75 E_i$

FIG. 4



The circuit for integrating (smoothing) the individual encoder output bits is shown in Fig. 2. The voltage E_i picked up by a brush such as A (shown as a switch arm) is applied across two equal resistors. A capacitor is connected across the lower resistor and a zener diode is back connected to the junction of the resistors. The resistor values preferably are high to limit the current drain from the source E_i and the capacitance value is selected to provide the desired averaging time constant. If the brush contact time exceeds 50% the voltage across the capacitor generally will be between $E_i/4$ and $E_i/2$. The zener diode therefore is selected to conduct at slightly more than $E_i/4$, providing the output bit "1".

The four outputs, A to D, may be connected to a conventional decoding matrix with 16 output lines for displaying the 16 compass point wind directions. The top half of Fig. 3 shows the first three lines of such a matrix. The bottom half of Fig. 3 shows an alternative matrix which provides 8 overlapping wind directions. Here the direction NNE is indicated by the simultaneous energization of the lines "N" and "NE".

To prevent damage to the power supply, brushes, or code disk in the event one of the brushes A to D is accidentally shorted to ground, the common voltage applied to the inner disk ring 22 may be ground rather than the supply voltage E_i . Fig. 4 shows the smoothing circuit in this case. The output bit "1" is produced when the brush circuit closure time is less than 50%. This causes an inversion of the cyclic code, with a corresponding change in the decoding matrices, shown in Fig. 5. Also the zener diode is selected to conduct at slightly more than $0.75 E_i$.

Under some weather service conditions it may be desirable to display the average wind direction in a 36-point format. A cyclic code having equal on-and-off distributions for this purpose is as follows.

DIR.	CODE	DIR.	CODE
36	000000	18	001111
01	100000	19	101111
02	110000	20	111111
03	111000	21	110111
04	011000	22	010111
05	001000	23	000111
06	001100	24	000011
07	101100	25	100011
08	111100	26	110011
09	110100	27	111011
10	010100	28	011011
11	000100	29	001011
12	000110	30	001001
13	100110	31	101001
14	110110	32	111001
15	111110	33	110001
16	011110	34	010001
17	001110	35	000001

APPARATUS FOR DISPLAYING AVERAGE WIND VANE OR OTHER SHAFT POSITION

H. H. Crouser

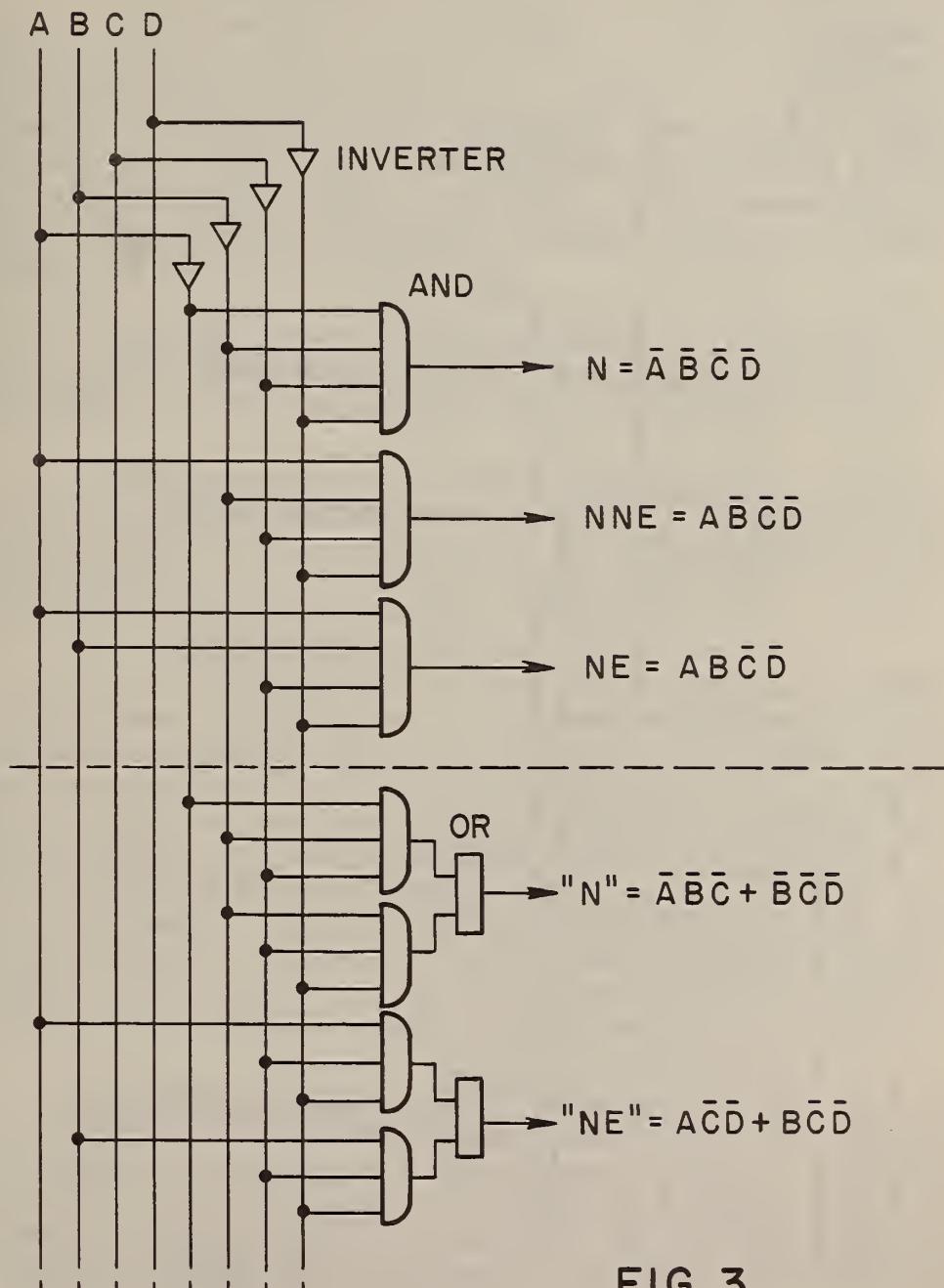


FIG. 3

APPARATUS FOR DISPLAYING AVERAGE WIND VANE OR OTHER SHAFT POSITION

H. H. Crouser

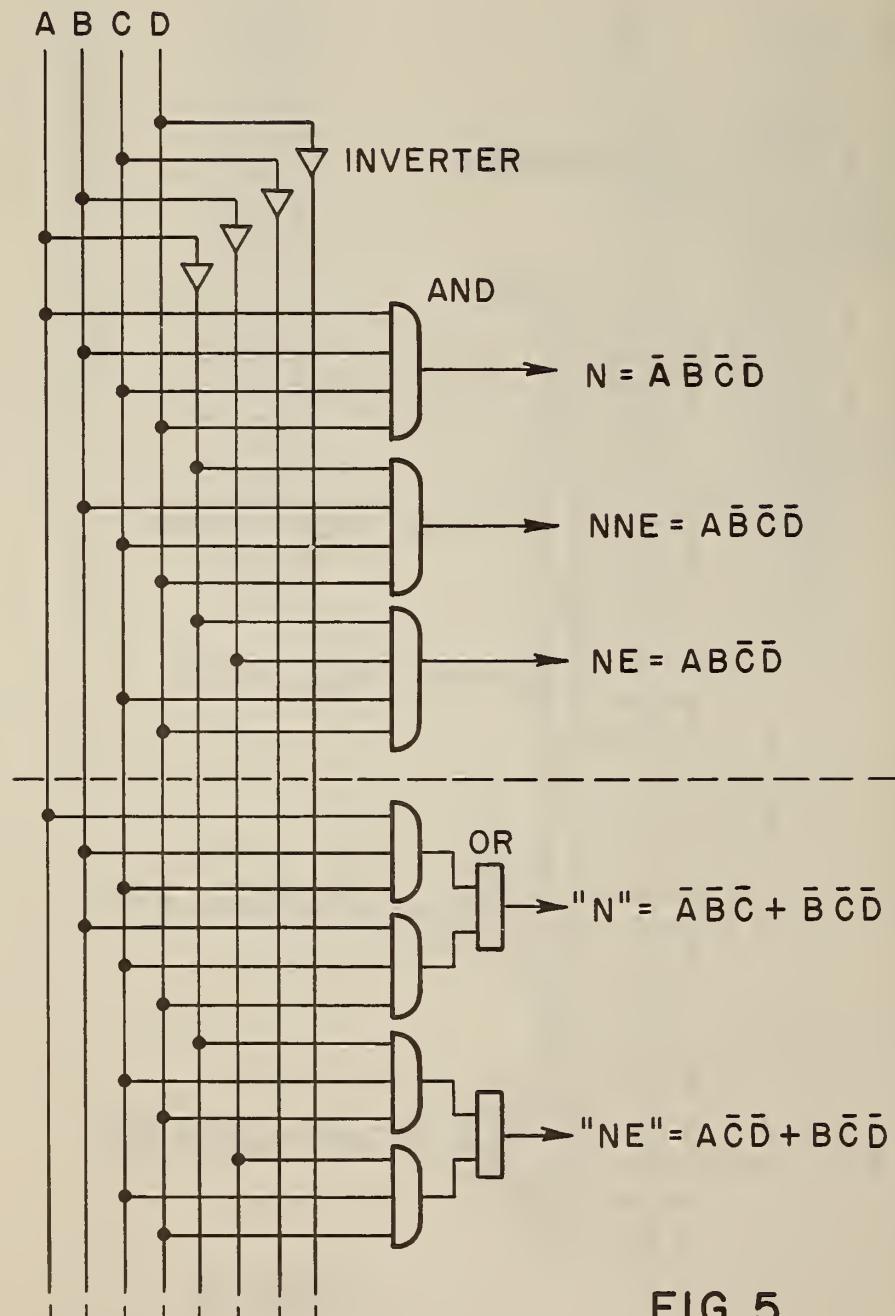


FIG. 5

DOCUMENT NUMBERING MACHINE RESPONSIVE TO A STAPLE IN A PRINT AREA FOR PRINTING IN AN ALTERNATE AREA

R. J. Varson and R. L. Johnson

In processing a multi-element separable document such as a tax return, it is common to print a document locator number (DLN) on all the elements to facilitate their location after the document is separated. The DLN can be automatically printed by a document feeding and numbering machine, provided the document does not contain a staple in the assigned print area. A staple will damage the print wheels, result in an illegible number, or cause an erroneous advance of the print wheels.

The purpose of this apparatus is to sense a staple in the print area of a document and delay the printing of the number so that it appears in an alternate area.

The apparatus is shown schematically in Fig. 1. It includes an inductive staple sensing probe (SP) 1 which is mounted ahead of the printer 24 in the document feed path. The sensing probe 1 drives a loaded oscillator (OSC) 2 which in turn strobes an AND circuit 4.

As a document is fed into the machine it interrupts a light beam impinging on a photocell (PC) 12, causing the photocell amplifier (PA) 13 to produce an output as long as the document is passing over the photocell 12. The leading edge of this output pulse is delayed by a delayed make circuit (DM) 14. The amount of delay is adjustable by the operator and serves to adjust the position of the first print on the document. The output of the delayed make circuit 14 is also used to trigger a pulse width control circuit (PWC) 3 which establishes a zone to be tested for staples by providing an enable to AND circuit 4.

If the sensing probe 1 detects a staple and therefore strobes AND circuit 4 while the latter is enabled by the output of the pulse width control circuit 3, the strobe signal will pass through and initiate a pulse width control circuit 5. This circuit 5 expands the staple pulse so that a staple detected at any time in the test zone will appear to exist during the entire test zone.

DOCUMENT NUMBERING MACHINE RESPONSIVE TO A STAPLE IN A PRINT AREA FOR PRINTING IN AN ALTERNATE AREA

R. J. Varson and R. L. Johnson

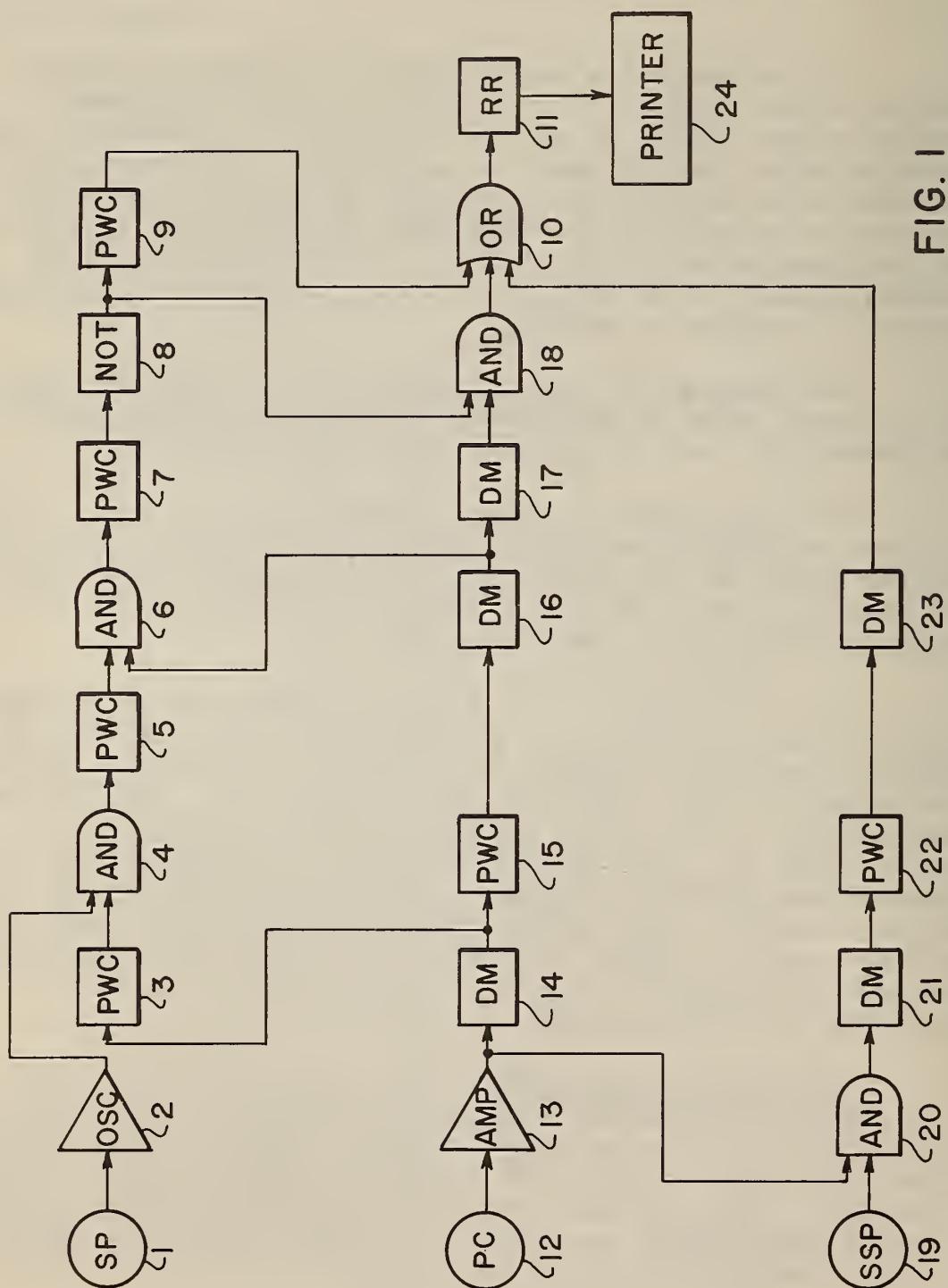


FIG. 1

The previously mentioned delayed make circuit 14 identified the location of the first print relative to the sense probe 1. Its output is applied to a pulse width control circuit 15 where it is extended beyond the first print location at the print head. The output of this pulse width control circuit 15 triggers a delayed make circuit 16 which delays it almost to the first print. Pulse 16 enables an AND circuit 6 at a time relative to the top of the document so that pulse 6 will not vary with the location of the staple. Pulse 6 triggers a pulse width control circuit 7 which is operator adjusted to set the alternate print location ("staple detected in first print location"). Pulse 7 is applied to an inverting circuit (NOT) 8. The restoration of pulse 8 at the trailing edge of pulse 7 triggers a pulse width control circuit 9 to produce a short pulse which passes through an OR gate 10 to operate a reed relay (RR) 11. The OR gate 10 is needed so that the reed relay 11 can be operated from more than one source to trigger the print circuits in printer 24.

If no staple pulse 1 occurred to coincide with enable 3 then circuits 4 through 9 as described above will remain inactive and pulse 16 will initiate a delayed make circuit 17 which delays it to approximately the middle of the test zone. Pulse 17 strobes an AND circuit 18 which is enabled by NOT circuit 8 if the alternate print logic was not activated by a staple. Pulse 18 passes through OR gate 10 to operate the reed relay 11 for a normal first print.

Should a second print be desired, a "select second print" circuit 19 enables an AND gate 20 allowing the photocell 12 to initiate a delayed make circuit 21. This circuit 21 is operator adjustable and delays the pulse to set the second print location and start a pulse width control circuit 22 which extends it beyond the second print location and triggers a delayed make circuit 23. This circuit 23 delays the leading edge to produce the print pulse. Pulse 23 passes through OR gate 10 and operates the reed relay 11 to trigger the printer 24.

Figs. 2 and 3 show two examples of the pulses produced by the circuits for documents of different sizes with staples in and after the test zone. The conditions for Fig. 2 are:

Minimum Page Length - Close Spaced
Minimum Delay First Print
Minimum Delay Second Print
First Document - Staple in Test Zone
Second Document - Staple after Test Zone

For Fig. 3 the conditions are:

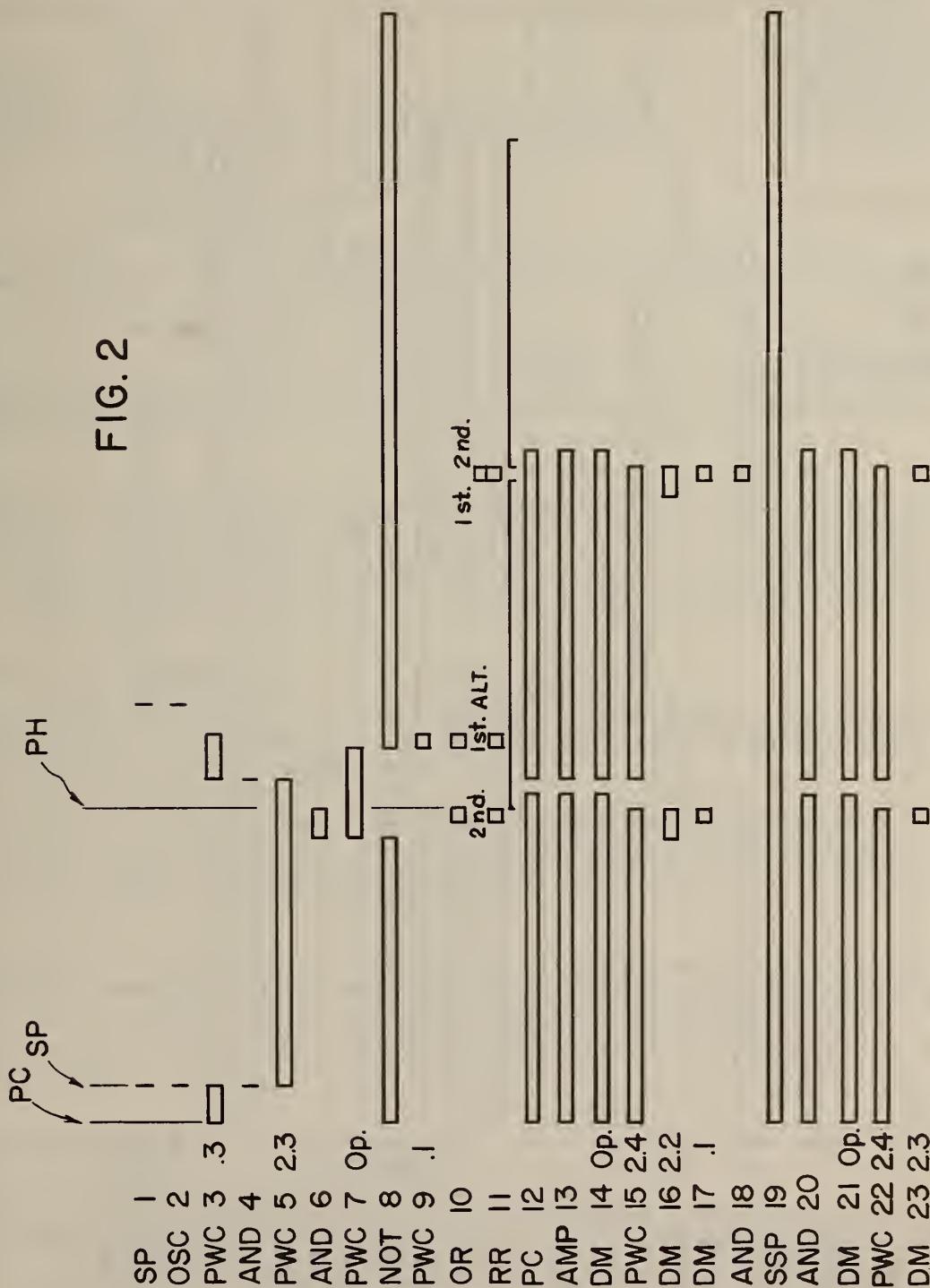
Five Inch Page Length - Wide Spaced
First Print $\frac{1}{4}$ Inch Below Top
Alternate Print One Inch After First
Second Print $\frac{1}{2}$ Inch from Bottom
First Document - Staple Early in Test Zone
Second Document - Staple Middle of Test Zone

The above-described apparatus is designed to print near the top of the document. Since staples are seldom encountered in other areas, the staple detection is applied only to the first print. However, the staple detection could be applied to the second print if desired. Also, the number of prints per document could be expanded.

DOCUMENT NUMBERING MACHINE RESPONSIVE TO A STAPLE IN A
PRINT AREA FOR PRINTING IN AN ALTERNATE AREA

R. J. Varson and R. L. Johnson

FIG. 2



DOCUMENT NUMBERING MACHINE RESPONSIVE TO A STAPLE IN A
PRINT AREA FOR PRINTING IN AN ALTERNATE AREA

R. J. Varson and R. L. Johnson

FIG. 3

PH

PC

SP

PC

1
OSC 2 .3
PWC 3 2.3
AND 4
PWC 5
AND 6
PWC 7 op.
NOT 8
PWC 9 !
OR 10
RR 11
PC 12
AMP 13
DM 14 op.
PWC 15 2.4
DM 16 2.2
DM 17 !
AND 18
SSP 19
AND 20
DM 21 op.
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